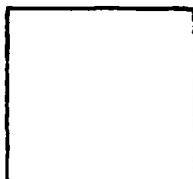# PHOTOGRAPH THIS SHEET

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HIGH PRESSURE OXIDATION
OF METALS

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MOLYBDENUM IN OXYGEN

by

Robert C. Peterson and
W. Martin Fassell, Jr.

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TECHNICAL REPORT
VI
September 1, 1954

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HIGH PRESSURE OXIDATION OF METALS

MOLYBDENUM IN OXYGEN

by

Robert C. Peterson and W. Martin Fassell Jr.

ABSTRACT

The temperature and pressure dependence of the reaction of molybdenum in oxygen were investigated from 525 to 700°C at pressures from one atmosphere to 47.6 atms. (700 psia). Molybdenum was found to oxidize linearly from 525 to 650°C at all pressures investigated. No dependence was observed from 525 to 550°C at pressures above one atmosphere. From 550 to 700°C the oxidation rate of molybdenum was dependent on oxygen pressure. The pressure dependence from 550 - 650°C may be expressed by $v = k \; \theta$ where k is the specific rate constant and

$$\theta = \frac{k_1 C_{O_2}}{1 + k_1 C_{O_2}}$$

where k_1 is the equilibrium constant for the adsorption of oxygen at the surface site of reaction. An unusual pressure sensitive transition from a linear rate to some type of protective rate was observed at 650 - 700°C.

INTRODUCTION

Numerous papers have been published on oxidation of molybdenum and molybdenum alloys.[1] Some of the most significant investigations on the rate of reaction of molybdenum in oxygen are those by Gulbransen and his associates. Gulbransen and Wysong[2] investigated the rate oxidations in oxygen using their microbalance technique from 250 - 450°C at low pressures (.076. 0 76, 7.6 cm. Hg.). They found that the metal follows the parabolic law under these conditions with an activation energy of 36.5 kcal/mole. Some deviation was noted toward a linear behavior at the higher temperatures.

Hickman and Gulbransen[3] investigated the types of films formed on molybdenum between 300 and 700°C by their electron diffraction technique. They found that in the low temperature range (300 - 400°C) MoO_3 becomes predominant in the surface oxide coating. Above 400°C, at the pressures used in their investigation, only MoO_2 was present. The absence of MoO_3 at high temperatures very possibly was due to the slow rate of conversion of MoO_2 to MoO_3 on the low oxygen concentration.

The present paper concerns the behavior of molybdenum from 500 to 700°C in oxygen at pressures ranging from one to 47.6 atmospheres (14.7 to 700 psia).

EXPERIMENTAL PROCEDURE

The equipment and the experimental technique used in this investigation have been reported in previous papers.[4,5,6] The metal samples used were sheared from 20 mil. molybdenum sheet (General Electric Company, Cleveland Wire Works) into small platelets approximately 1.5 inches X 0.5 inches. Spectrographic analysis of the molybdenum metal indicated the following impurities present 0.0001% Cu., 0.001% Ni. No analyses were made for absorbed or dissolved gases in the metal.

Prior to oxidation each sample was abraded lightly with No. 1 metallographic paper washed in acetone and dried. Each individual observation was made at constant temperature and pressure.

EXPERIMENTAL RESULTS

The results to be described are based on the experimental observations of over 100 molybdenum samples in oxygen from 500 to 700°C at absolute oxygen pressures from one to 47.6 atmospheres (14.7 to 700 psia.). Molybdenum was found to oxidize according to the linear mechanism from 500 to 650°C at all pressures investigated. Figure 1 shows a typical weight gain versus time curve in this region. From 650 to 700°C deviations from linearity were found in the sense that the rate of oxidation decreased markedly with increasing time at the lower pressures. At higher pressures, however, this anomolous deviation from linearity was reduced or completely eliminated. For example, at 650°C and at atmospheric pressure the rate of oxidation decreases with increasing time.
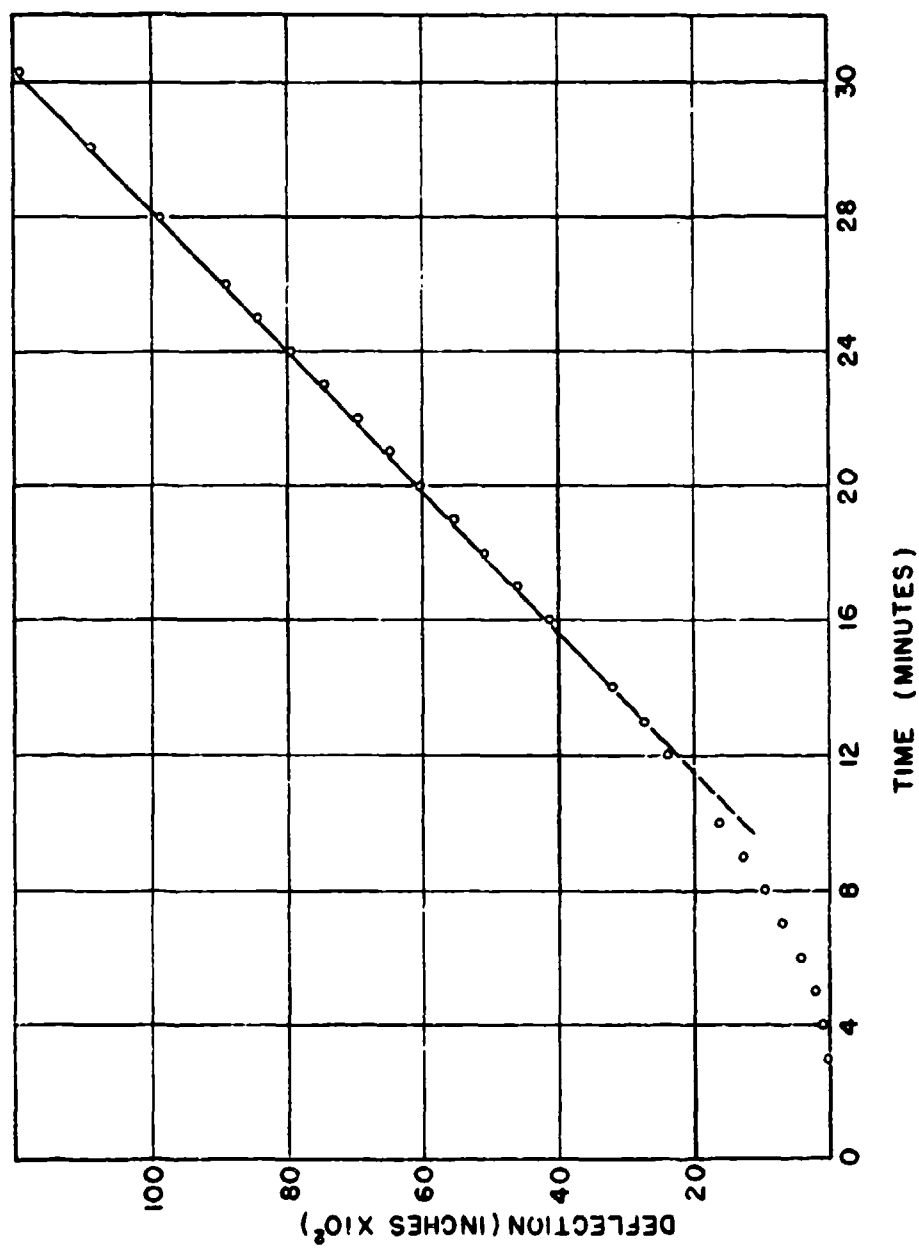
Fig. I. TYPICAL LINEAR OXIDATION RATE OF MOLYBDENUM IN OXYGEN AT 600°C. AND 200 psia.

If the pressure is increased to 50 psia. the metal returns to its linear behavior. At 700°C it was necessary to increase the pressure to 400 psia. to cause the reversion to the linear rate. This peculiar behavior of molybdenum at 700°C with respect to oxygen pressure is shown in Figure 2. It should be mentioned at this point that to the authors' knowledge this is the first time that the reversion from a linear rate to rate law of higher order, i.e., parabolic. cubic or logarithmic. has been observed.

At temperatures below 500 to 525°C no variation in the rate of oxidation with respect to oxygen pressure was observed at the pressures investigated. At 550°C the rate of oxidation of molybdenum becomes dependent on the oxygen pressure. The experimental and theoretical oxidation rates as a function of oxygen pressure at various temperatures are shown in Figures 3 to 8.

DISCUSSION OF RESULTS

The experimental results presented above clearly demonstrate the following facts concerning the oxidation behavior of molybdenum under the imposed conditions. (1) molybdenum oxidizes linearly from 500 to 650°C at all pressures investigateu, (2) at temperatures from 550 to 700°C the oxidation rate is a function of the oxygen pressure while below this temperature no pressure effects were observed (3) from 650 to 700°C the metal shows a peculiar transition from some type of protective behavior at low pressures to linear behavior at high pressures; (4) at any given temperature from 550 to 650°C the rate of oxidation approaches a limiting rate as the pressure is increased.

X-ray diffraction studies made on the oxide films formed on the metal in situ indicated that the films were highly oriented by virtue of the fact that a number of the normal diffraction lines of MoO_3 were totally absent. However, when the oxide films were removed from the metal substrate and finely ground. the resulting d/n values correspond closely to the reported values for MoO_3. The d/n values obtained for the oxide in situ and after removal are given in Table I. The slight shift in the d/n values of the oxide in situ suggests that possibly some residual strains were present in the oxides at room temperature. In no case were diffraction patterns observed for the lower oxides of molybdenum by X-ray techniques.
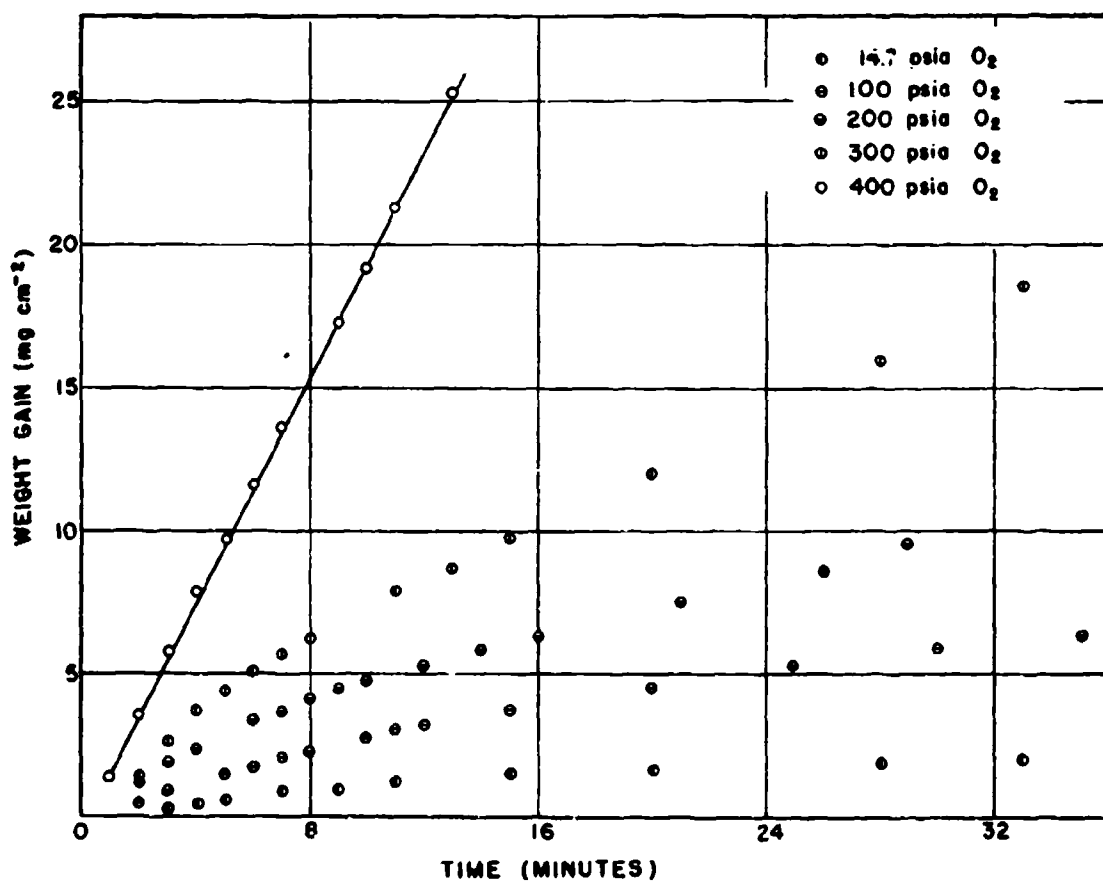
Fig. 2. OXIDATION RATE OF MOLYBDENUM IN OXYGEN AT 700° C.
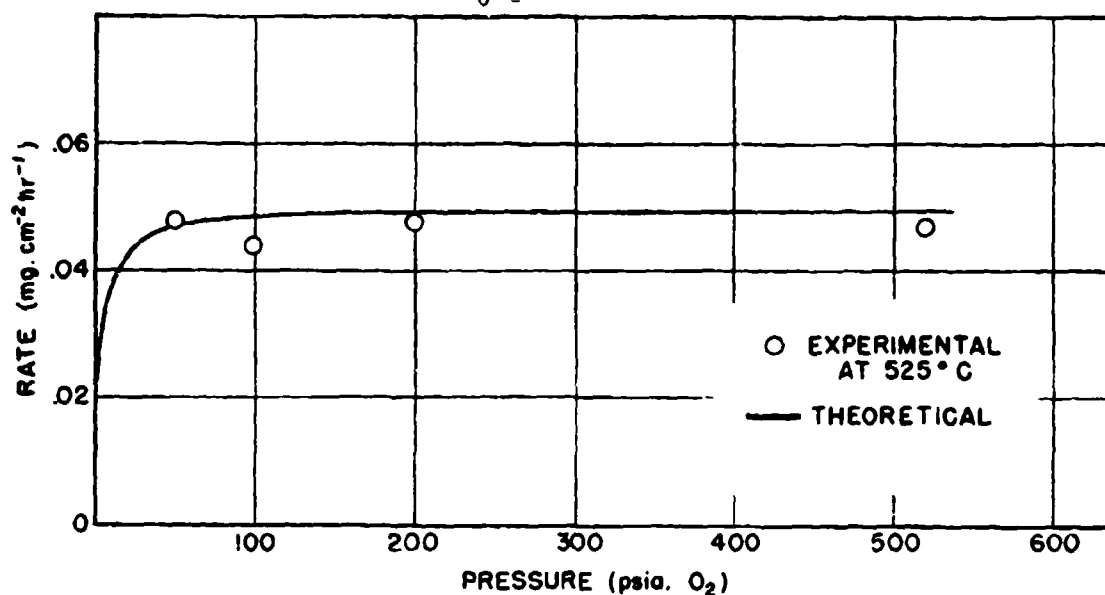SHOWING THE TRANSITION FROM PROTECTIVE TO LINEAR
RATES AT HIGH PRESSURES

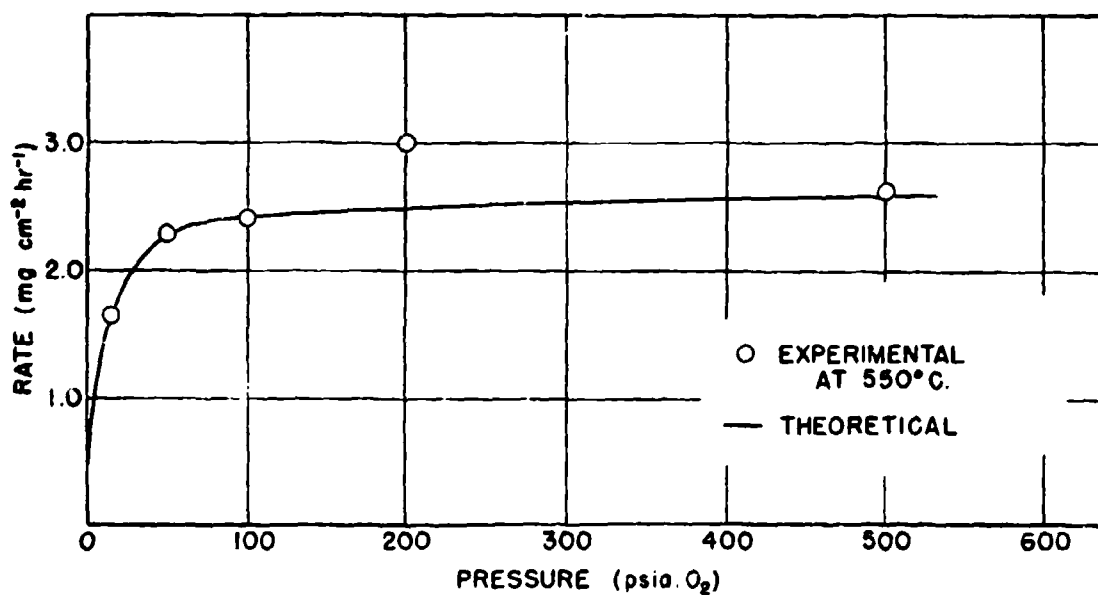Fig. 3. OXIDATION RATE OF MOLYBDENUM versus OXYGEN PRESSURE

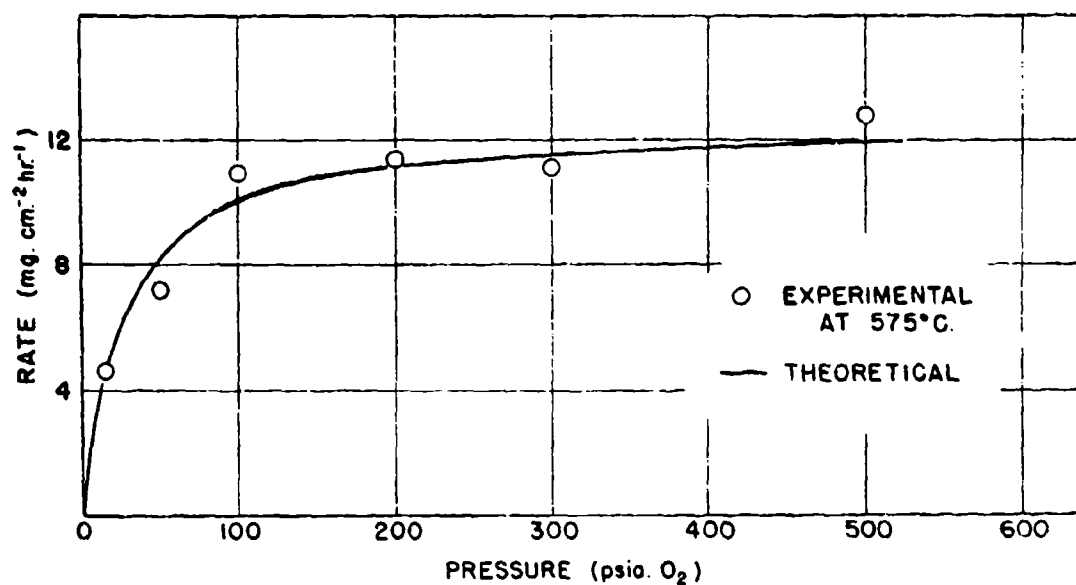

Fig. 4. OXIDATION RATE OF MOLYBDENUM versus OXYGEN PRESSURE

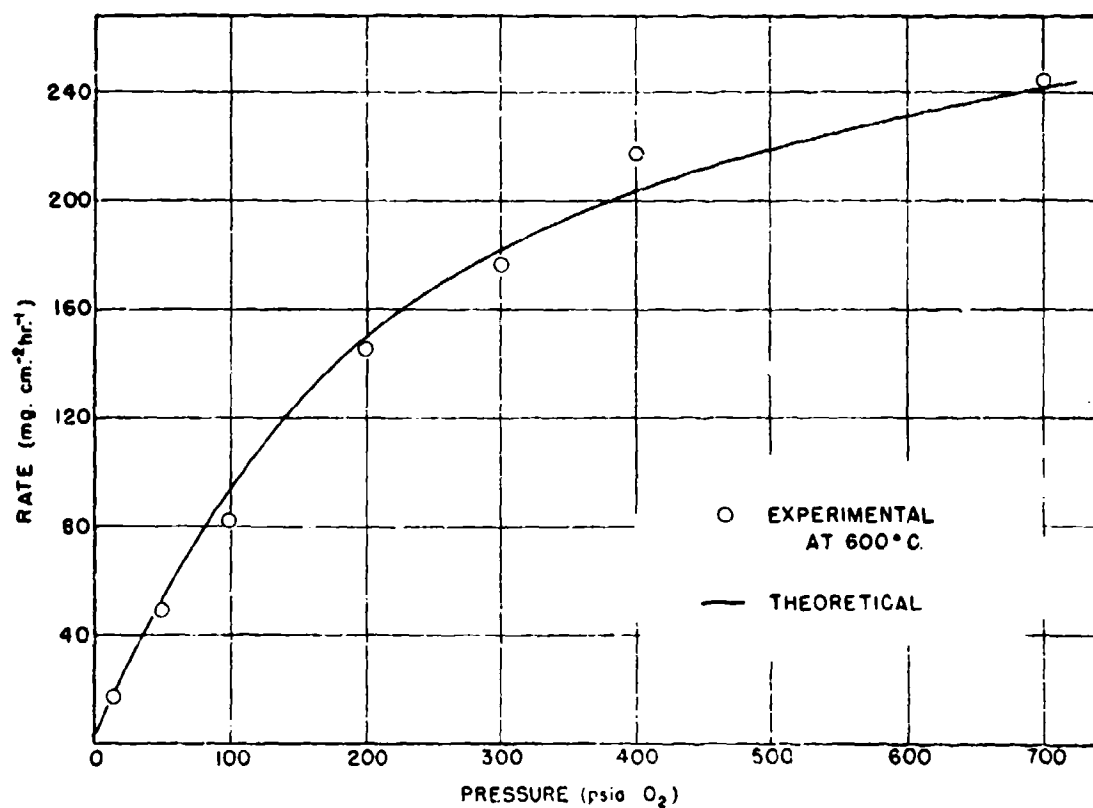Fig. 5. OXIDATION RATE OF MOLYBDENUM versus OXYGEN PRESSURE

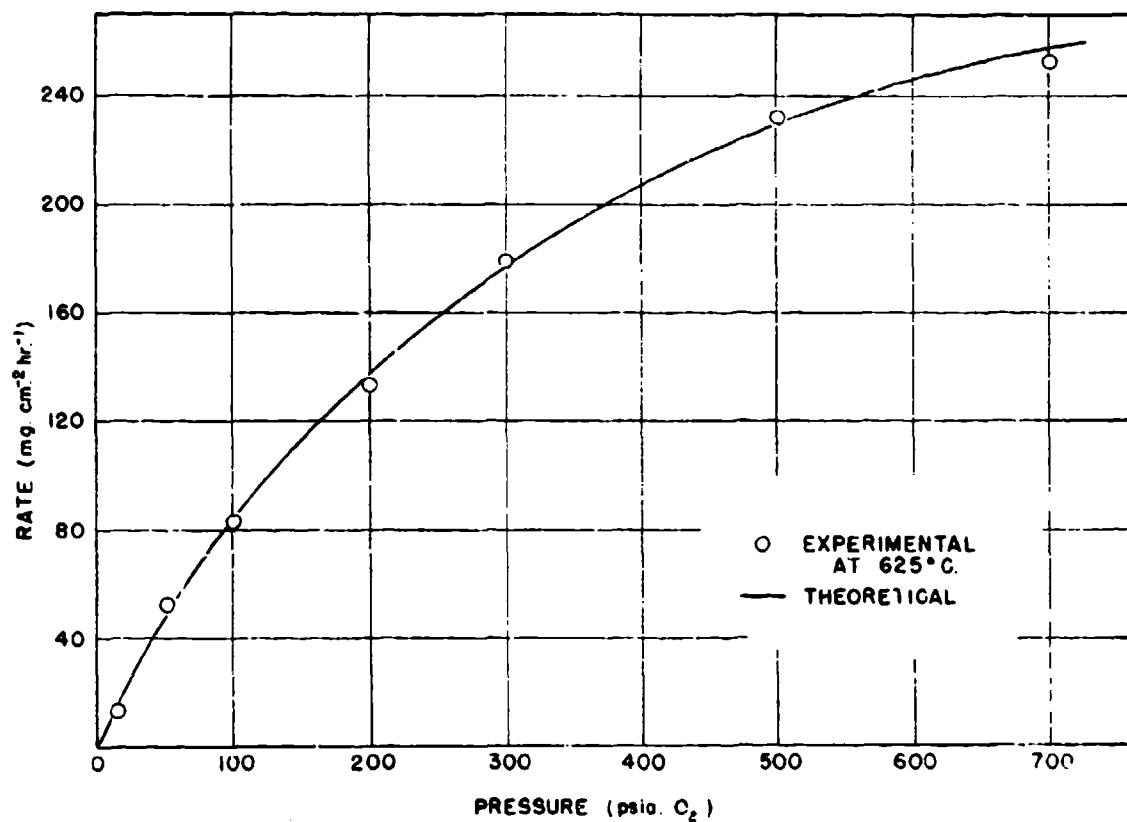Fig. 6. OXIDATION RATE OF MOLYBDENUM versus OXYGEN PRESSURE

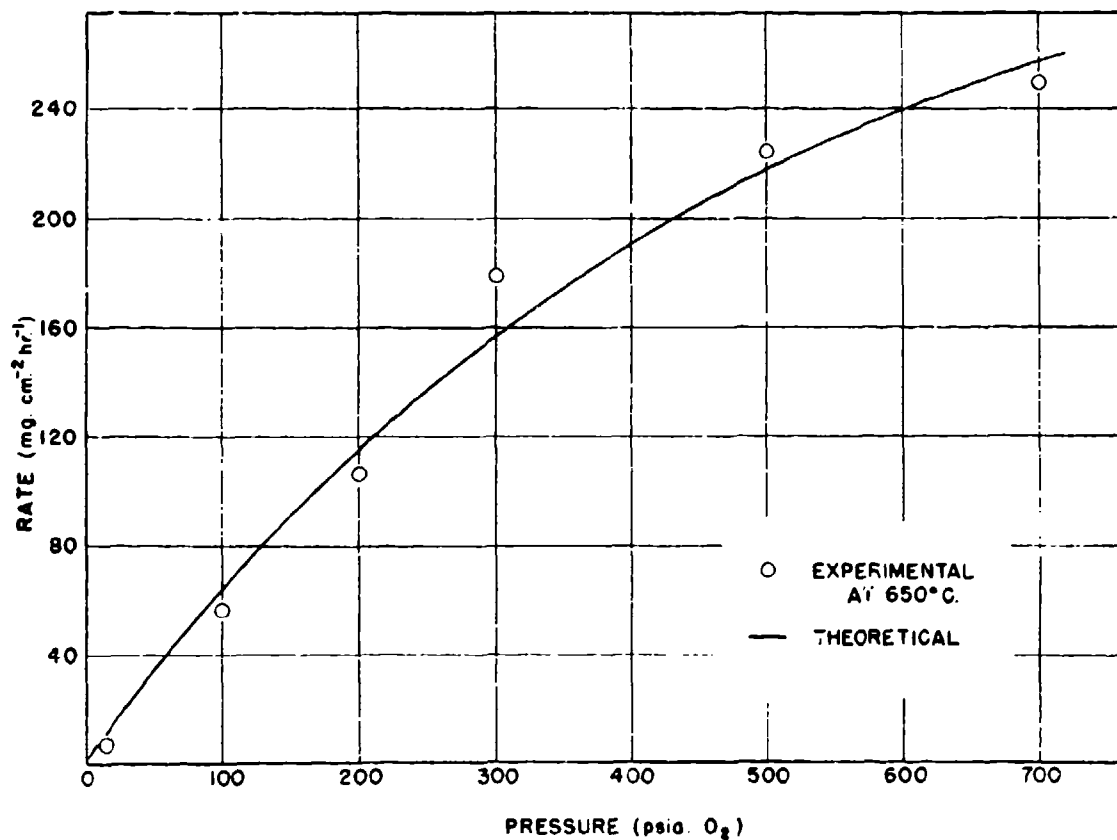Fig.7. OXIDATION RATE OF MOLYBDENUM versus OXYGEN PRESSURE

Fig.8. OXIDATION RATE OF MOLYBDENUM versus OXYGEN PRESSURE

TABLE I

COMPARISON OF THE LATTICE PARAMETERS OF THE
OXIDE FILMS FORMED ON MOLYBDENUM METAL

OXIDE FILM IN SITU d/n	GROUND OXIDE FILM d/n	ASTM MoO_3 d/n	ASTM MoO_2 d/n
4.1329	7.5634	6.9	3.415
3.7385	6.8624	3.80	2.811
3.3759	5.8176	3.46	2.693
2.1709	3.4530	3.25	2.424
2.1462	3.2433	3.00	2.177
1.9649	2.5493	2.66	1.844
1.9489	2.3037	2.52	1.722
1.8039	1.9569	2.30	1.708
1.4269	1.7338	2.26	1.695
1.2914	1.5679	2.13	1.531
	1.5302	1.97	1.467
	1.3840	1.85	1.402
		1.73	1.390
		1.70	1.301
		1.67	1.237
			1.215

═══ STRONG

─── MEDIUM

NO LINE   WEAK

Some question may exist concerning the possible sublimation of the MoO_3 during oxidation. A number of samples were oxidized to completion and a comparison was made between final and stoichiometric weight corresponding to MoO_3. No significant loss of the oxide could be detected up to and including 700°C. In addition no sublimed crystals were observed on the interior of the equipment.

Numerous attempts were made to secure data at 725°C. At this temperature ignition of the metal invariably occurred, preceded by 'self heating' of the metal sample. In fact even at 700°C some indications of self heating were observed but the furnace was sufficiently responsive to correct this condition, making rate measurements possible.

The results presented in the above figures and the conventional Arrhenius plot shown in Figure 9 indicate the complexity of the oxidation of molybdenum. In addition it has been shown by others that at low temperatures (below 450°C) molybdenum follows the parabolic law.
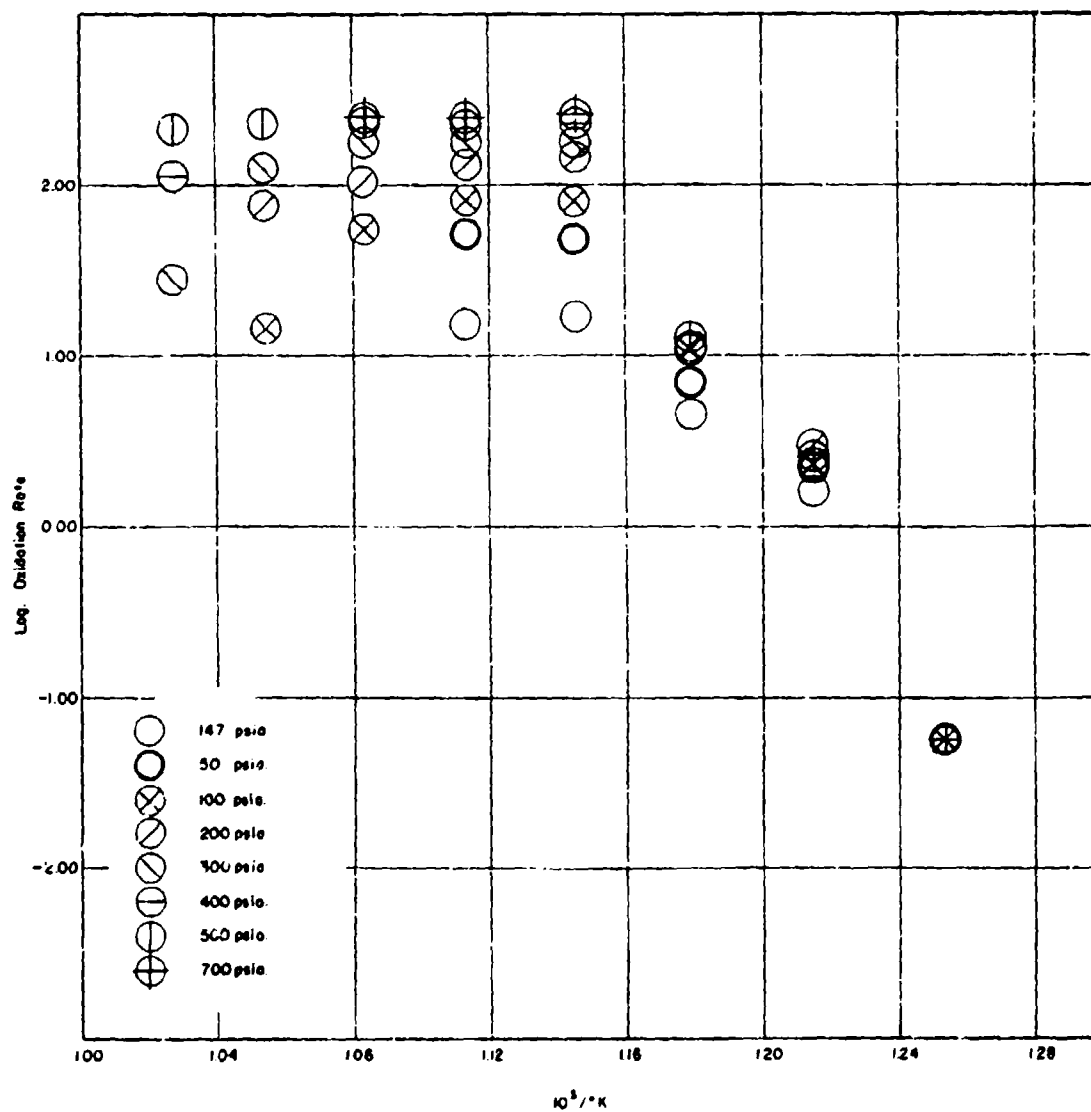
Fig. 9. ARRHENIUS PLOT OF THE EXPERIMENTAL OXIDATION RATES OF MOLYBDENUM IN OXYGEN

The present results indicate a shift to a linear rate at intermediate temperatures, which incidentally has become a fairly familiar pattern of many metals, followed by a pressure sensitive reversion to a pro- tective mechanism at higher temperatures (650 to 700°C).

The exact nature of this protective mechanism is at present still unknown. Repeated attempts to apply the conventional protective type laws have not produced entirely satisfactory results. It may be significant to mention the fact that such a shift was predicted pre- viously[7] on the basis of a rather qualitative consideration of the relative rates at which the film forming oxide was produced and the rate at which it could be assimilated in the already present film. Briefly a possible explanation for the reversion to the protective mechanism for molybdenum is as follows: In the intermediate tempera- ture region (from 500-650°C) where the linear rate law is followed, the rate of oxide formation is greater than the rate at which this oxide can be assimilated in existing crystallites by some type of growth me- chanism. Therefore a high probability exists that nuclei will form, resulting in extremely small MoO_3 crystals throughout the oxide film, resulting in a relatively porous, non-protective film. As the temperature increases the rate of oxidation will increase according to the usual exponential relationships. Likewise, from the theoretical considera- tions of sintering, it is known that the growth process will increase exponentially. The question then becomes one of the relative effect of temperature on these two exponential functions. While quantitative data are lacking on MoO_3, it might be expected that as the melting tem- perature of MoO_3 is approached, the rate of crystal growth will become large with the possibility that the oxide film could now 'fit in' the new oxide formed to the existing oxide crystallites. The shift from a pro- tective mechanism to a linear mechanism with increased pressure at 700°C may then be considered to occur because of the increased rate of oxidation at higher pressures in this temperature region. Further work is in progress on this transition because of its possible theoretical implications.

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THEORETICAL

The linear equation usually given to describe the oxidation of a metal is $w = kt$, where w is the weight gained and k is the rate. The value of k under the experimental conditions which are described in the paper is a function both of temperature and of the pressure of the gas phase of the system. The temperature dependence is usually given in the forms of the Arrheniu equation

$$V = A e^{-\frac{E}{RT}} \tag{1}$$

where V is the experimental rate, A the action constant. E is the activation energy, and R and T are the gas constant and absolute temperature. Fig. 9 shows a plot of the logarithm of the rate versus $1/T$. The large deviation from a straight line due to the effect of pressure is immediately obvious.

The linear equation can be re-written in the form,

$$w = \prod_i c_i f(T) \tag{2}$$

where the rate is implicitly a function of the product of the concentrations at the site of the reaction of the reacting species and of the temperature. Assuming that $f(T)$ is given by the Eyring (8) equation for the absolute rate of reaction, the question then becomes one of determining $\prod_i c_i$ in terms of the effective concentration of oxygen in the gas phase.

The rate is now in the form,

$$V = \prod_i c_i \mathscr{K} \frac{kT}{h} e^{-\frac{\Delta H^{\ddagger}}{RT}} e^{\frac{\Delta S^{\ddagger}}{R}} \tag{3}$$

where

$$\mathscr{K} \frac{kT}{h} e^{-\frac{\Delta H^{\ddagger}}{RT}} e^{\frac{\Delta S^{\ddagger}}{R}}$$

is the specific rate constant as given by Eyring.

In the present investigation it was found that the rate versus pressure curves shown in Figs. 3 to 8 could be fitted almost exactly with a type I Langmuir Adsorption Isotherm. This indicates that the concentration term $\prod_i c_i$ in Eq. (3) is proportional to θ. the fraction

of the available surface sites covered by adsorbed oxygen. In terms of θ Eq. (3) becomes,

$$V \cdot k_o \; \theta \; \mathcal{R} \; \frac{hT}{h} \; e^{-\frac{\Delta B^{\ddagger}}{RT}} \; e^{\frac{\Delta S^{\ddagger}}{R}} \tag{4}$$

where k_o is a proportionality constant with the dimensions of ML^{-2} and includes the correction factor for the true surface area involved in the oxidation reaction.

An expression for θ in terms of the gas phase concentration of oxygen can be obtained by considering the following equilibrium reaction between an absorption site, S_a, and O_2 in the gas phase,

$$S_a + O_2 \rightarrow S_a - O_2 \; (ads). \tag{5}$$

The fraction of sites covered, θ, is proportional to $S_a - O_2$ (ads) and the fraction of bare sites, $1 - \theta$, to S_a. The equilibrium constant for Eq. (5) is then,

$$k_1 \cdot \frac{\theta}{1 - \theta} \cdot \frac{1}{f_{O_2}} \cdot e^{-\frac{\Delta F_1}{RT}} \tag{6}$$

where f_{O_2} is the fugacity of O_2 in the gas phase and ΔF_1 is the free energy of adsorption of oxygen on the surface. The standard state for this system is chosen as that which corresponds to one-half surface coverage and a pressure of one atmosphere. Oxygen can be considered an ideal gas under the experimental conditions herein described so that Eq. (6) can be rewritten,

$$k_1 \cdot \frac{\theta}{1 - \theta} \cdot \frac{1}{C_{O_2}} \tag{7}$$

where C_{O_2} is now the effective concentration of O_2 in the gas phase. Solving for θ, we obtain,

$$\theta \cdot \frac{k_1 \, C_{O_2}}{1 + k_1 C_{O_2}} \tag{8}$$

Substituting Eq. (8) in Eq. (4) and making the usual assumption that $\mathcal{R} = 1$, we have

$$V \quad k_o \left[\frac{k_1 \, C_{O_2}}{1 + k_1 C_O} \right] \frac{kT}{h} e^{-\frac{\Delta B^{\ddagger}}{RT}} e^{\frac{\Delta S^{\ddagger}}{R}} . \tag{9}$$

TABLE II

$T^\circ C$	$T^\circ K$	$\dfrac{10^3}{T}$	k_1	$\log k_1$
525	798	1.2531	240	2.38
550	823	1.2151	128	2.107
575	848	1.1792	39.6	1.598
600	873	1.1455	4.5	.653
625	898	1 1136	2.75	.440
650	923	1.0834	1.35	.130

At constant temperature Eq. (9) can be written as,

$$V = k_0' \left[\frac{k_1 C_{O_2}}{1 + k_1 C_{O_2}} \right] \tag{10}$$

and this in turn can be cast into linear form,

$$\frac{1}{V} = \frac{1}{k_0 k_1 C_{O_2}} + \frac{1}{k_0} \tag{11}$$

from which k_0 and k_1 can be determined by plotting reciprocal rate versus reciprocal concentration as is shown in Figure 10. Table II gives the values of the equilibrium constant of adsorption. k_1. obtained in this manner.

Figure 11 shows a plot of $\log k_1$ versus reciprocal temperature from which the heat of adsorption. ΔH_1. of O_2 on molyhdenum was found be 61.0 kcal/mole of oxygen with an entropy of adsorption ΔS of 36.4 entropy units. Both ΔH_1 and ΔS_1 are considerably larger in this case than the corresponding values for the adsorption of oxygen which was involved in the oxidation of tantalum under similar conditions.[8]

From Eq. (9) it is evident that a plot of

$$\log V \left[\left(\frac{k_1 C_{O_2}}{1 + k_1 C_{O_2}} \right) \frac{kT}{h} \right]^{-1} \quad \text{vs.} \quad \frac{1}{\circ K}$$

should yield a straight line. Figure 12 shows such a plot. In contrast to Fig. 9 where a large spread in the experimental values due to neglecting the surface concentration of O_2 is evident, the experimental points now fall reasonably well along two straight lines. The length of the veritcal line within the circles in Fig. 12 indicates the experimental spread of the rates at various pressures.
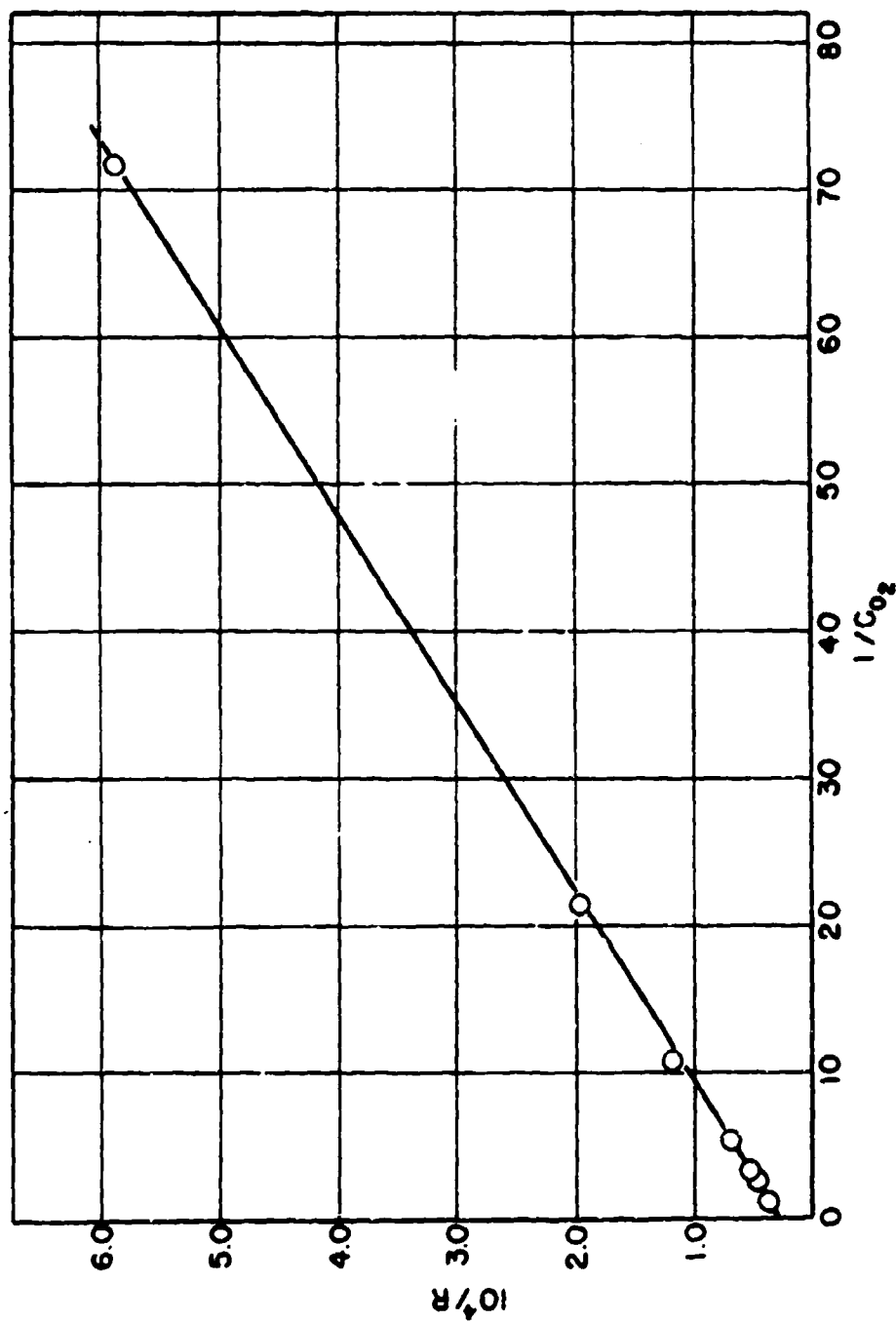
Fig. 10. TYPICAL CURVE USED TO EVALUATE k_1 AND k_0 IN EQUATION
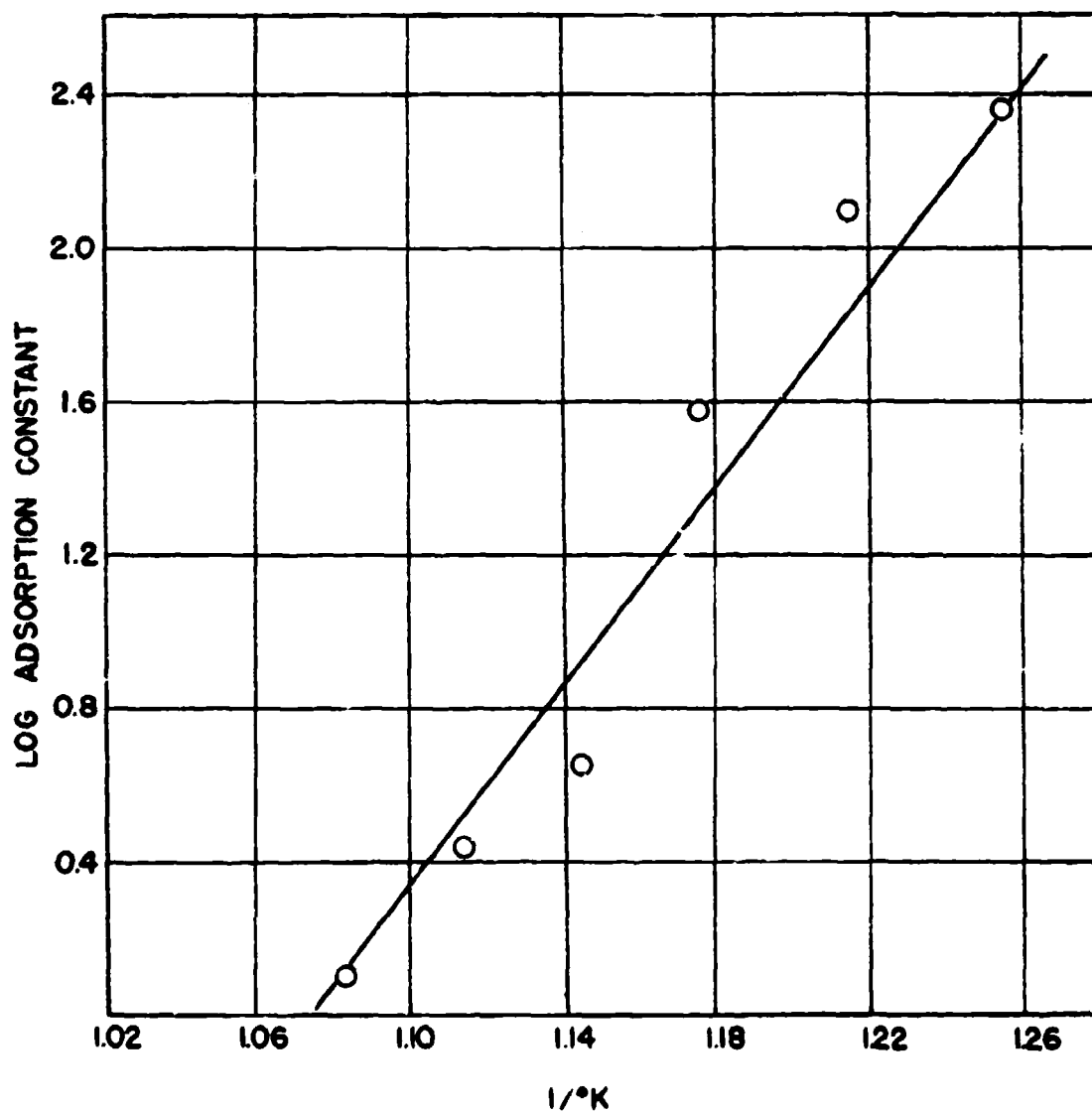
Fig. 11.   RELATIONSHIP BETWEEN EQUILIBRIUM
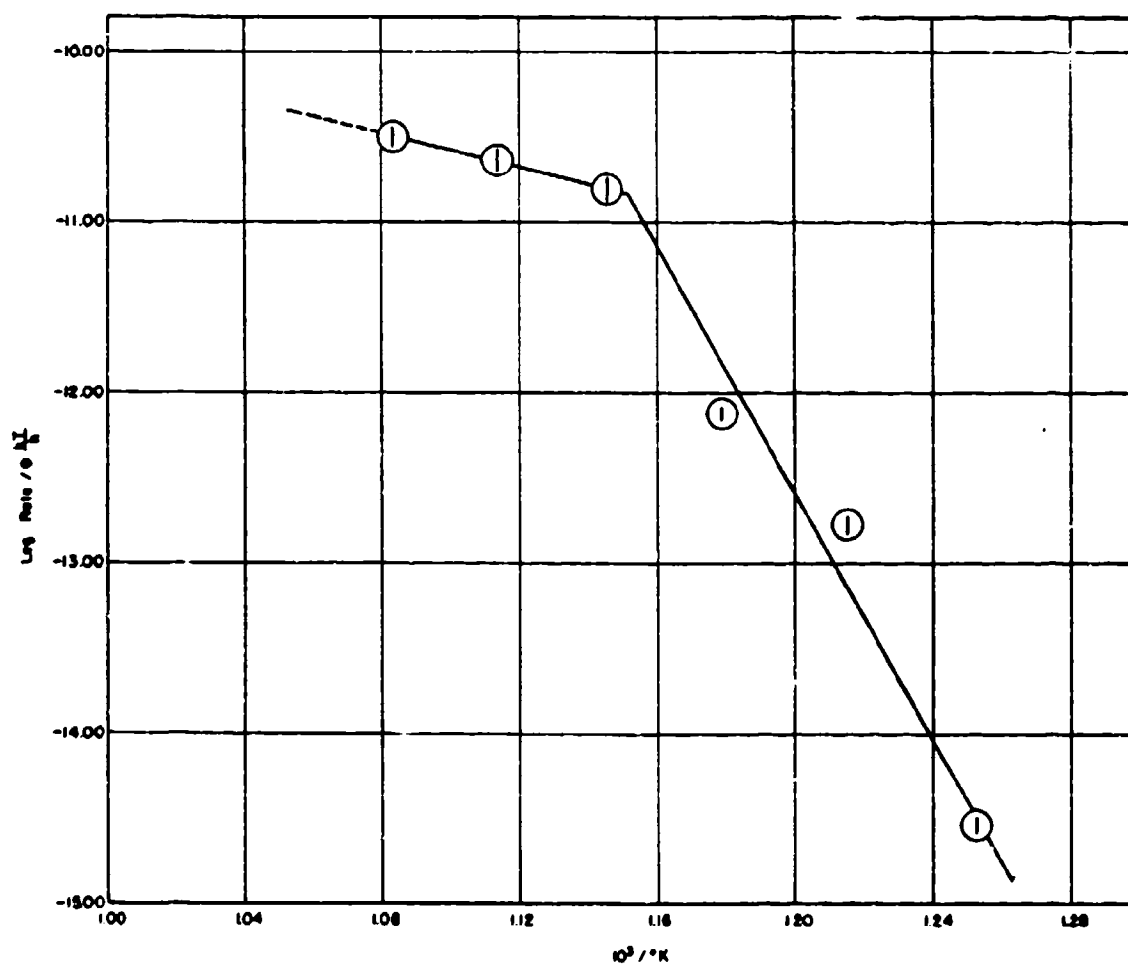CONSTANT OF ADSORPTION AND
TEMPERATURE

Fig. 12. TOTAL ABSOLUTE RATE OF REACTION OF MOLYBDENUM IN OXYGEN versus
RECIPROCAL TEMPERATURE

The heat of activation. $\Delta H^{\ddagger}$, as determined from Fig. 12 in the temperature region from 525 to 625°C is 165 kcal/mole. In the temperature range from 625 to 700°C $\Delta H^{\ddagger}$ is approximately 23.3 kcal/mole. The extremely large value of $\Delta H^{\ddagger}$ in the first case above is indicative of the great sensitivity of the rate of reaction of molybdenum in oxygen to temperature changes. At equivalent concentrations of oxygen the rate at 625°C is greater by a factor of ten thousand than the rate at 525°C.

SUMMARY

In conclusion. the facts concerning the oxidation behavior of molybdenum in oxygen from 500 to 725 C at pressures ranging from 1 to 47.6 atmospheres pressure are as follows.

1. Molybdenum oxidizes according to the linear law from 500 to 650°C at all pressures investigated.
2. No pressure dependence of the oxidation rate was found from 500 to 525°C.
3. From 550 to 700°C the oxidation rate is sensitive to oxygen pressure. In this region the reaction rate may be expressed by the equation

$$ V = k_0 \, d' \left(\frac{k_1 C_{O_2}}{1 + k_1 C_{O_2}} \right) e^{-\frac{\Delta H^{\ddagger}}{RT}} e^{\frac{\Delta S^{\ddagger}}{R}} $$

    where $\Delta H^{\ddagger}$ has a value of 165 kcal/mole from 525 to 625°C and 23.3 kcal/mole from 625 to 700°C.
4. A peculiar pressure sensitive transition has been observed from 650 to 700°C wherein the mechanism shifts from some unknown type of protective behavior at low pressures to a linear mechanism at high pressures. It is believed that this is the first experimental observation of a shift from a linear rate to a protective rate with increasing temperature.
5. The oxide formed on the metal under the experimental conditions used was MoO_3.

ACKNOWLEDGEMENT

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